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Semiautomatic Modulus of Rupture Tester for Operation in Vacuum or Inert Atmospheres

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The construction and operation of a semiautomatic modulus of rupture tester for determining the modulus of rupture up to 2400°C in either vacuum or inert atmospheres are described. One of the unique features of the apparatus is its ability to test up to 18 specimens without being reloaded. This paper includes a description of the auxiliary equipment used in conjunction with the tester and a typical curve of modulus of rupture against temperature obtained with this tester.

AUTHOR

INTRODUCTION

THE modulus of rupture or flexural strength for a beam of rectangular cross section with freely supported ends is defined by the equation

$$S = \frac{3}{2} PL / bh^2,$$

where S is the modulus of rupture in units of force per unit area, P is the force at rupture applied to the beam at midspan, L is the span between supports, b is the width of the beam, and h is the height of the beam.

The modulus of rupture is a useful criterion for comparison of the relative strengths of brittle materials up to temperatures at which the materials deform plastically. Oxides, carbides, borides, and many other ceramic-type materials are commonly tested in this manner. The ease with which the test specimens can be prepared, the speed with which the tests can be carried out, and the relative simplicity of the test equipment required for room-temperature testing, among other factors, account for the popularity of this testing method. High-temperature testing for the modulus of rupture has, in common with other testing procedures, the disadvantage that time is required for heating and cooling the furnace as well as for loading and unloading of test specimens. It is the purpose of this paper to describe a modulus of rupture tester for high-temperature use in which a large number of specimens can be tested in the same run without opening the furnace. In addition, the furnace used in conjunction with the tester is described.

FURNACE

The high-temperature vacuum furnace used is shown schematically in Fig. 1. This vacuum furnace is induction heated with a graphite susceptor and is mounted between the crosshead and the table of a 20 000-lb capacity, Tinius Olsen Electromatic tensile machine. The furnace is supported by four legs of adjustable height, which are bolted to the floor. Except for the top and bottom plungers, no part of the furnace touches the tensile machine.

The 6½-in.-i.d., 16-in.-long graphite susceptor and the carbon susceptor extensions rest on the water-cooled

bottom plate and are kept in position by means of pins (not shown). The susceptor is located inside a 10-in.-i.d., 32-in.-long fused silica tube, which fits into annular grooves in the water-cooled top and bottom plates. Silicone rubber gaskets in these grooves seal the heating chamber vacuum tight. Carbon black insulation fills the annular space between the susceptor assembly and the silica tube. Glass or graphite cloth backed by a segmented graphite ring prevents the carbon black insulation from being blown out during evacuation of the furnace. The furnace connects to the vacuum system through a port in the water-cooled manifold that is welded to the top plate. Four thick tie rods between the top and bottom plates align the furnace and ensure a rigid assembly. Power is provided through a water-cooled induction coil wrapped around the silica tube.

The modulus of rupture tester rests on a carbon block insulator, which, in turn, rests on the water-cooled bottom plunger. Both the tester and the carbon block clear the susceptor assembly by about ⅛ in. A Teflon bellows assembly between the bottom plunger and the bottom plate provides vacuum tightness without appreciable effect on the vertical motions of the bottom plunger and prevents it

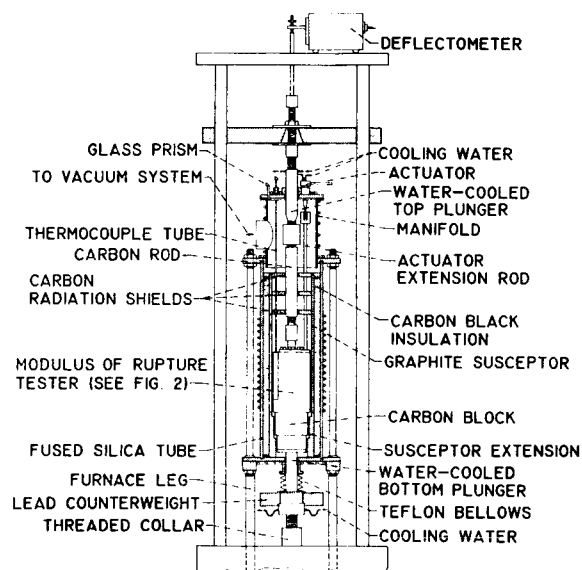


FIG. 1. High-temperature vacuum furnace.

from rotating. A threaded collar allows vertical adjustments of the bottom plunger. A lead counterweight balances the atmospheric pressure on the plunger when the furnace is under vacuum.

The force required to break the test specimen is transmitted from the crosshead to the tester through the water-cooled top plunger and a carbon rod. Couplings between the top plunger and the carbon rod and between the carbon rod and the tester complete the force-transmitting link. The flat lower portion of the actuator fits loosely into a slot in the adapter, which is attached to the actuator extension rod. The actuator assembly allows transmission of rotational motion to the tester without interfering with its vertical motions.

A thermocouple tube with a tungsten-rhenium thermocouple and beryllium oxide insulators can reach inside the tester to determine specimen temperatures. The thermocouple tube enters the furnace through a Conax fitting, and the tube can be raised above the carbon radiation shields for operation at temperatures above the range of the thermocouple assembly. Temperatures can also be determined with an optical pyrometer by sighting the specimen through a glass prism in the furnace cover. A system of vacuum valves (not shown) in the tube where the prism is located allows removal and cleaning of the prism while the furnace is in operation.

The water-cooled furnace cover is provided with O-rings for sealing all its openings vacuum tight. This cover is provided with an internal flange and with external guide pins for accurate centering and location.

Crosshead motions are sensed by a deflectometer connected to the top plunger through an adjustable-length link. The crosshead motion and the load are recorded on the tensile machine recorder.

Up to about 1200°C vacuums of less than 0.01 μ Hg are readily obtained. As the temperature increases, the pressure gradually builds up to about 10 μ Hg at 2800°C because of evaporation of volatile matter from the carbon parts. This pressure is expected to decrease with use as the volatile matter is driven out of the carbon parts.

The maximum power required to hold the furnace at 2800°C is about 20 kW.

Calibration of the tensile machine plus the furnace system by means of dead weights showed that, in addition to the tensile machine percent error, frictional forces in the furnace and variations in atmospheric pressure introduce an error of less than ± 2 lb in determining the actual loads applied to the test specimens.

This furnace design lends itself to a high degree of versatility. By the use of various internal parts, the apparatus may serve as a hot press, tensile tester, or compression tester in addition to its primary design purpose as a modulus of rupture tester. So far, our particular furnace has been used for modulus of rupture testing and as a hot

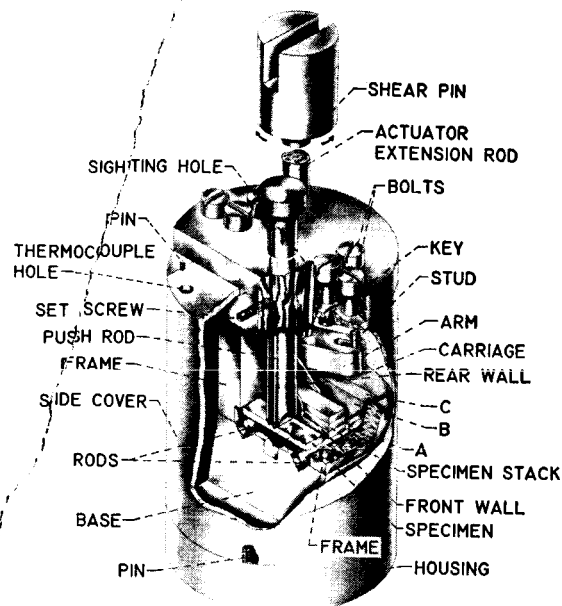


FIG. 2. Semiautomatic modulus of rupture tester.

press. During hot pressing, a load maintainer in the tensile machine keeps the load at any desired level, and the crosshead motions can be followed with the deflectometer to determine the specimen densification with time, pressure, and temperature.

Metered argon or helium can be used instead of a vacuum as an atmosphere or for rapid cooling below 1500°C. Overheating of the silica tube above this temperature precludes the use of argon or helium except at low pressures.

MODULUS OF RUPTURE TESTER

The modulus of rupture tester to be described here is of the centerpoint-loading type and differs from the familiar type of modulus of rupture tester in that it is provided with a magazine for storage of specimens, a means of moving the specimens into position, and a means for removing the broken specimens. This modulus of rupture tester is shown in Fig. 2.

The tester is made entirely of graphite. The housing o.d. is 6 in. and the height is $8\frac{3}{4}$ in. The lid is secured to the housing, and at the same time centered, by means of graphite bolts. The load is applied to the specimen through a push rod, which slides up and down the center hole in the lid. This push rod is prevented from rotating by a set screw whose end fits into a groove in the push rod.

The specimens rest on $\frac{1}{2}$ -in.-diam rods located in V grooves in the base. These rods are fastened to the base by means of pins (not shown). The V grooves in the base are accurately machined to provide a 2.000-in. span between the centerlines of the rods.

A carriage can slide back and forth on a track cut in the base. The carriage is actuated by rotating the actuator extension rod, the motion being transmitted to the carriage

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through an arm and a stud. The arm is secured to the actuator extension rod with a key. The stud is fastened to the carriage and extends below the carriage to fit into a slot (not shown) in the base. The size of this slot limits the backward motion of the carriage, so as to prevent the carriage from touching the susceptor.

The magazine for storage of specimens is formed by the rear wall, the front wall, and the two side frames. These side frames are provided with vertical grooves on which the front and rear walls of the magazine fit. The side frames are held in place by long bolts that pass through the lid and the base and screw into the bottom of the housing. The rear wall is provided with an opening at the bottom that just allows passage of the horizontal step B of the carriage (Fig. 2). The bottom piece in the stack of specimens in the magazine rests on the rods. In this position, the bottom piece is too high to pass through the opening in the rear wall, but it can pass through a bottom opening in the front wall when pushed by vertical surface A of the carriage.

The side cover is held in place by means of pins. This cover prevents the broken specimen pieces from entering the annular space between the tester and the susceptor and, at the same time, provides a "blackbody" enclosure for more accurate optical temperature measurements through the sighting hole in the lid. There is a hole in the side cover through which the thermocouple tube can be inserted.

The specimens used in this particular setup are prismatic bars of rectangular cross section 2.35 in. long, 0.500 in. wide, and 0.250 in. high. As already mentioned, the span between the $\frac{1}{2}$ -in. rods on which the specimens are broken is 2.000 in. The distance between the side frames, which limits the lateral motion of the test specimens, is 2.40 in. Specimens higher or lower than 0.250 in. can be used provided the bottom opening of the front wall is enlarged or shortened accordingly.

The magazine is filled with test bars either through the rear by removing the lid and the rear wall or through the bottom opening in the front wall with the aid of a spatula.

The device operates as follows. With the tester installed in the furnace as shown in Fig. 1 and with the specimen in the position shown in Fig. 2, the specimen is loaded at midspan by the push rod, which is actuated by the cross-head, until the specimen breaks. The motion of the cross-head is reversed until it returns to its original position, as determined by reading the recorder chart. As the actuator is rotated, the vertical surface A of the carriage pushes the bottom test bar in the magazine through the bottom opening of the front wall. This new specimen pushes the broken specimen to the edge of the rods, from where it falls to the bottom of the housing. At the end of its forward travel, vertical surface C of the carriage will be touching the rear wall, while the new test bar will be in its test posi-

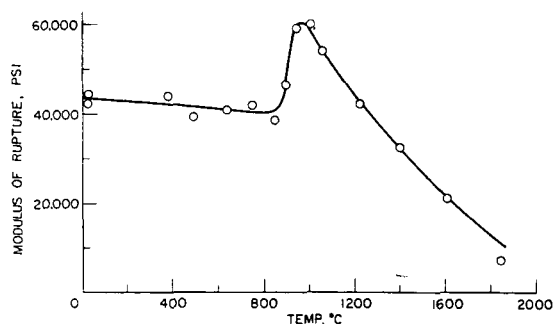


FIG. 3. Plot of modulus of rupture against temperature for titanium-modified zirconia.

tion right under the push rod and ready for breaking. While the carriage was pushing the specimen into test position, the bottom specimen in the stack dropped on horizontal surface B of the carriage but was prevented from moving with the carriage by the front wall. When the motion of the carriage is reversed, this test bar drops on the rods and is ready for a repetition of the sequence described above.

The capacity of the magazine is 18 pieces of the size mentioned above.

The apparatus described above has been operated at a total force of 1000 lb without ill effects and at temperatures up to 2400°C without noticeable dimensional changes. Full-load runs with graphite test specimens were carried out. No trouble was encountered with the graphite specimens up to the maximum test temperature of 2400°C. The graphite test specimens (GS grade, National Carbon Company) were prepared without particular care about dimensions or surface conditions since the only objective of this test was to determine the suitability of the feeding mechanism. Plastic yielding in this graphite was readily observed in the curve of crosshead motion against load in the recorder. The first indication of plastic deformation was obtained in the 2200°C test. This plastic deformation was later confirmed by determining the radius of curvature of the broken specimens.

Runs were also made with titanium-modified zirconium oxide specimens. These specimens could be fed easily by the actuating mechanism at all temperatures up to 1400°C, at which temperature the specimens started sticking to each other. Subsequent runs in which the top specimens in the magazine were separated from each other with small pieces of 5-mil tungsten wire made it possible to operate the feeding mechanism up to 1800°C, at which temperature the specimens started sticking to the graphite rods and to the carriage. These troubles were caused by evaporation of titanium metal from the test specimens followed by reaction of the titanium with the graphite parts. Considering the reactivity of titanium, the device was operating under really disadvantageous conditions. This kind of trouble is not expected to occur with the great majority of ceramic materials.

A typical curve of modulus of rupture against temperature obtained with the aid of this tester is shown in Fig. 3. This curve was obtained in an 8-h run in which about 20 min were allowed for each specimen to reach an equilibrium temperature. This curve is included for illustration purposes only, and data regarding specimen preparation, curve shape, and so forth will be included in a separate study. Regarding the unusual shape of the curve, it will suffice to say that the zirconia in this material undergoes a phase transition from monoclinic to tetragonal which starts at about 950°C on heating.

In cases where the thermal history of the specimen may influence the modulus of rupture values, the tests can be carried out "on the run" since the temperature gradient in specimens of the dimensions used is small and is not expected to alter the modulus of rupture significantly. In the great majority of cases, however, the materials used are either sintered or hot pressed well above the maximum testing temperature, and soaking at temperature before testing should not significantly affect the results since grain growth, sintering, and so forth are strongly temperature dependent and should be negligibly small compared with the same effects at the sintering temperature.